Materials Science Division Project Safety Review
Safety Analysis Form (03/08)

Date of Submission	03/01/10	FWP No.:	58405

Project Title	TEM/SEM Specimen Preparation Work in Laboratories 212/DL126 and 212/EL205 (part of the Electron Microscopy Center) This Safety Analysis Form (SAF) supersedes previous version of 4/22/2008.

Is this a (check one) new submission [ ] renewal [ ] supplemental modification [X]

Principal Investigator(s) Jon M. Hiller

Other Participants (excluding administrative support personnel) EMC staff and various EMC users

(Attach participant signature sheet)

Project dates:	Start:	03/01/10	End:	Open-ended

This form is to be completed for all new investigations or experimental projects that are conducted in MSD laboratories, and for all ongoing such projects that undergo significant change from their original scope of work, or where there has been an addition of a potentially new hazard not covered in the original review. It is not intended to be used for office work, routine maintenance activities, or administrative tasks.

Experimental work may not be performed until the project safety review has been completed, procedures have been approved, and the work has been authorized (ESH Manual 21.2.3).

The completed form and all supporting documentation is to be submitted to the MSD ESH Coordinator by the principal investigator with sufficient advance notice and information to allow a project safety review prior to the beginning of the experiment. The information will be reviewed by the Division Director, members of the MSD safety review team, and by outside experts (if appropriate) for unresolved safety, health, and environmental issues associated with the proposed work. The principal investigator may be asked to resolve outstanding issues through consultations with the safety review team before the work begins. The information submitted will be reviewed by an independent review team, and final approval will be granted by the Division Director.

This form must be accompanied by a participant signature form once work has been authorized.

The principal investigator must be familiar with the responsibilities of a lead experimenter and the general requirements of the experiment safety review in the Argonne ESH Manual, section 21.2.

Useful references:
Argonne ESH Manual: http://www.aim.anl.gov/manuals/eshman/
Argonne Waste Handling Procedures Manual: http://www.aim.anl.gov/manuals/whpm/
MSD Chemical Hygiene Plan: http://www.msd.anl.gov/resources/esh/
Material Safety Data Sheets: https://webapps.inside.anl.gov/cms/msds/

Table of Contents[1]:

List of Attachments:

DL 126 Lab Hazards
DL 126 Lab Rules
VCR Ion Mill SOP
PIPS SOP

Plasma Cleaner SOP

[1] To update table of contents, right/command click inside table; from resulting contextual menu select "Update field", then "Update page numbers only"

1. Scope of Project (ISM Core Function 1)

1.1 General Description

Provide a general overview description of the project. While scientific background is important, concentrate on an operational description that focuses on the experimental work done in the laboratory.

This project provides and manages laboratories (building 212, rooms DL126 and EL205) dedicated to the production of specimens for electron microscopes in the Electron Microscopy Center (EMC). The laboratory will be used by EMC staff and EMC users (scientists from Argonne National Laboratory and other institutions).

Activities include – but are not limited to – mechanical cutting, grinding, polishing, and dimpling; cleaning samples with organic solvents; chemically etching metals and ceramics; electropolishing; ion-milling with argon or nitrogen, cleaning/etching/ashing with argon or oxygen plasmas; microtoming; using various epoxies for gluing samples and creating samples for microtoming.

1.2 Modules of Project

Describe the various components that make up this project. Components can be pieces of equipment or specific hazardous or complex tasks within the project that require special training to use or perform safely. Indicate locations, even if the project consists of only one component. Indicate custodians for major equipment. Attach designs, drawings, or other useful descriptive material.

Jon Hiller is the custodian for all sample preparation equipment. Russell Cook is the alternate.

Components (major equipment):

- Dimplers
- Ion Mills
- Polishing wheels
- Diamond wheel and wire saws
-
- Electropolishing apparatus
- Disc cutters
- Sputter coater and carbon evaporator
- Microtome (EL205)

1.3 Project Limits

Define the range of samples, chemicals, physical conditions that you consider covered under this project review. For chemicals and samples include either specific cases that are considered extremely hazardous, e.g. silane, HF, etc., or general classes such as reactive metals, oxidizers, etc. In some cases it may be useful to define the envelope by specifically excluding certain hazard categories.

Specifically excluded under this SAF are:

1. Specimen preparation of radioactive materials
2. Specimen preparation of materials rated biosafety level 2 or higher
3. Use of hydrofluoric and perchloric acids

Prohibited tasks while working alone:

- Electropolishing, etching, or chemical polishing with acids/bases
- Mixing corrosives (acids and bases) to form a working solution

No high hazard processes are allowed without the presence of the laboratory manager (Jon Hiller).

Work in EL205 is limited to using the Reichert-Jung Ultracut E microtome.

Users are not allowed to store their finished samples in DL126 and EL205. This limitation eliminates some potential hazardous wastes. EMC staff are allowed such storage.

2. Hazard Analysis (ISM Core Function 2)

2.1 Hazard List

Hazard/Issue	Yes	No	Unknown
Does the proposed work, as you perceive it, intrinsically contain the following safety, health, or environmental issues or concerns?			
Chemical Hazards			
Use of toxic chemicals	X		
Use of flammable chemicals	X		
Use of carcinogenic chemicals	X		
Generation of hazardous or toxic wastes	X		
Use of explosive or highly reactive chemicals		X	
Use of strong acids or bases	X		
Use of carbon monoxide gas		X	
Use of hydrogen gas (above 4% concentration)		X	
Use of perchloric acid or perchlorate salts		X	
Use of hydrofluoric acid		X	
Nanomaterials			
Nanoparticles dispersible in air	X		
Nanoparticles dispersible in liquids	X		
Biological Hazards			
Work with Biosafety Level 2 or above[2]		X	
Radiological Hazards			
Use of radioisotopes (see section 6)		X	
Exposure to ionizing radiation (excluding radioisotopes)		X	
Generation of radioactive wastes		X	
Physical Hazards			
Use of Class III or Class IV lasers		X	
Use of cryogenic fluids	X		
Use of high magnetic fields		X	
Use of high voltage or high amperage equipment	X		
Electrical work on energized equipment (>50V)		X	
Operation of equipment at high vacuums	X		
Operation of equipment at elevated pressures	X		
Use of compressed gases	X		
Operation of equipment at high temperatures	X		
Hazardous Working Environments			
Working in areas with high noise levels		X	

[2] Requires review by Institutional Biosafety Committee

Potential exposure to climatic extremes		X	
Working at elevated heights		X	
Entering confined spaces		X	
Use of self-contained breathing apparatus or respirators		X	
Work in areas of mechanical hazards	X		
Other (explain)[3]			
Sharps	X		

2.2 Hazard Details

For all the hazards checked Yes or Unknown, provide specific details, including locations (unless obvious from 1.3). For highly hazardous or energetic chemicals, indicate specific chemicals, quantities used. For physical hazards, give quantitative details (e.g., voltages for electrical hazards, power and wavelengths of lasers).

Hazard	Detail
Toxic Chemicals	See attached sheet of chemicals
Flammable Chemicals	See attached sheet of chemicals
Carcinogenic Chemicals	See attached sheet of chemicals
Hazardous Waste	Located in the satellite waste accumulation area # 21200006.
Acids/Bases	Stored in the cabinets under 2 separate fume hoods
Nanomaterials	1. Diamond and aluminum oxide slurries in suspension. 2. Some users may request to bring small quantities (micrograms) of nanoparticles into DL126 to use for making SEM or TEM specimens in the hoods. The EMC generally prohibits this activity.
Cryogenic Fluids	Liquid nitrogen is used in two ion mills (about 2 liters each).
High Voltage/Amperage	Voltage ranges from 50 V to10 kV (ion mills). Current ranges from ~1 μA to ≤ 50A (carbon coater).
High Vacuums	Equipment under vacuum is at 10 to 10^{-4} Pa
Elevated Pressure	Nitrogen at ≤ 9 psi is available at several locations. Medium pressure air at ≤ 90 psi is available at several locations.
Compressed gases	The EMC provides argon at ≤ 24psi to ion mills, a sputter coater, and plasma cleaners/etchers. Small cans of pressurized R134a are used for cleaning specimen surfaces. Small cylinders of oxygen and an oxygen mixture (10% O_2, 90% Halocarbon-14) are used rarely in the SPI Plasma Prep etcher.
High Temperatures	A hot plate is typically used at 100C to 150C, however it can reach 250C. A vacuum oven is typically used at 70C. All other sources are generally not accessible (water chiller components, ion gauges)
Mechanical Hazards	Saws and disc cutters have protective shields. The high-speed saw also has a switch that shuts down the saw when the shield is removed. Vacuum seals and flanges could create potential pinch hazards for fingers.
Sharps	Tweezers, scalpels and razor blades

2.3 Waste Produced

Describe types and expected quantities of wastes produced by this project (also see 3.6 for the handling of these wastes). Address all applicable major classes (nonhazardous, hazardous/chemical, radioactive, mixed) and the specific types within these classes. Also include wastes that derive from the future decommissioning of equipment (e.g., transformer oils, accumulated debris in reaction chambers) and termination of projects (leftover chemical inventory, samples, materials).

[3] See ESH Manual 21.3 Appendix A for a more comprehensive list of potential hazards

1-2 gallons/year of waste pump oil ("special waste"). 1 gallon/year of acetone, 1 gallon/year of methanol, and 1 gallon/year of other alcohols used for cleaning. 2 cubic feet/year of towels soaked with oil or acetone or methanol. Less than 1 liter of both acid and base waste is generated per year.

The participants are responsible for removing their specimens and materials from the EMC.

2.4 Effluents and Emissions

Describe types and expected quantities of materials disposed into the water drains (effluent). Include process water if the amount disposed is unusually large (e.g., constantly running water). See ESH Manual 10.4 for rules regarding disposal of chemicals into the waste water stream. Consult with the building manager for stricter rules due to the condition of drain pipes.

All users of the DL126 are instructed that only water is disposed of in the laboratory sink/drain. All users are instructed on how to log and place chemical waste in the Satellite Waste Accumulation Area during their training (see section 3.4).

Describe types and expected quantities of gases, aerosols, and other volatile materials emitted to the atmosphere through the ventilation system (incl. hoods)

Roughing pump exhaust gases are piped into the fume hood exhaust system through a HEPA filter in 212/DL126 and not into the laboratory spaces. This is a continuous process while instruments are under vacuum.

Are all components of this project considered "bench scale research" (NEPA)? Yes X No ____
[Limited in any single experiment, measurement, or test to 5 gal. or 5 lbs of hazardous material, or 1 lb of extremely hazardous material (40 CFR 355)]

2.5 Interaction With Other Projects

Describe possible interactions with other projects (or separate components within the same project) that are carried out in the same or adjacent spaces. Are there incompatibilities of hazards that need to be mitigated by spatial separation or staggered times of operation (e.g. lasers vs. other work in laser access controlled area). Does this project introduce major hazards into a building that are not covered under the current Building Emergency Plan (Bldg. 223 emergency plan on MSD intranet, http://www.msd.anl.gov/resources/esh/ , confer with building managers for other buildings)?

The MSD SAF 20003 project (Electropolishing TEM Samples Using Perchloric Acid) requires a temporary rope barrier to keep non-participants away from the hazards that it introduces to 212/DL126. When that project is active, the east hood is unavailable for any other work.

3. Hazard Control (ISM Core Function 3)

3.1 Design Features and Engineering Controls

For all hazards present, describe the design features and engineering controls applied to control the hazards. Engineering controls include enclosures and barriers that cannot be removed without the use of tools, interlocks, ventilation, software controls, etc. Engineering controls are possible and should be first line of control for all hazard classes (chemical, physical, electrical, biological, radiological).

Hazard	Hazard Control
Toxic Chemicals	Two hoods are available in DL126 for work with these chemicals.
Flammable Chemicals	Two hoods are available in DL126 for work with these chemicals. These chemicals are stored in a flammable hazards cabinet in

	DL126.
Carcinogenic Chemicals	Two hoods are available in DL126 for work with these chemicals.
Hazardous Waste	Two hoods are available in DL126 for work that generates hazardous waste. A flammable hazards cabinet is present in DL126 for storage of flammable hazardous waste.
Acids & Bases	Two hoods are available in DL126 for work with these chemicals.
Nanomaterials	Two hoods are available in DL126 for work with these materials.
Cryogenic Fluids	Cryogenic transfer dewars are available. Ion mills have dewars and transfer lines designed into their systems.
High Voltage/Amperage	Engineering controls have been built into the instruments using safety switches, shielding, and other safeguards to protect users from high voltage/amperage.
High Vacuums	Engineering controls have been built into the instruments using safety switches, shielding, and other safeguards to protect users from high vacuum systems. All systems have built in pressure relief via gravity mounted o-ring seal ports or flanges.
Elevated Pressure & Compressed Gases	Large gas cylinders are secured to the wall with cylinder clamps and straps. Small gas cylinders are secured to a bench with cylinder clamps and straps. The argon regulator is fitted with an ASME-approved relief valve with a lift pressure of 60 psig.
High Temperature	The hot plate is located on a lab bench on the south side of the lab away from all flammable chemicals located on the north side of the lab. The vacuum oven has a latched door to prevent users from touching the heated interior.
Mechanical Hazard	All the instruments have some type of shielding from moving parts and are transparent to the user.
Sharps	All usable sharps are stored in a closed container. Broken or unusable sharps are stored in another container for disposal.

3.2 Procedural Controls

For complex hazardous tasks, describe how the hazards are controlled by the work procedure. E.g., specific order of tasks, verification of instrument readings, required use of special tools, and the like.

For complex tasks, hazards are controlled by requiring users to follow step-by-step standard operating procedures (SOPs) or manufacturers' instructions. Deviating from the normal SOP's or manufacturers' instructions will require approval from the EMC director and a separate SAF.

Toxic and flammable chemicals:
Users are made aware of the chemical toxicity/flammability. Users will read the appropriate MSDS.

Carcinogenic chemicals:
Typically the carcinogenic chemicals are stored in the sample preparation lab. Carcinogen containers are labeled and designated carcinogen areas are posted in accordance with the provisions described in Section 4.5.7. of Argonne ES&H manual. Any work with these chemicals is done in the fume hood. The user is instructed to attend the ANL carcinogenic class and update their JHQ. Under no circumstances will a carcinogenic chemical be milled, ground, sliced or diced in the sample preparation lab.

Generation of hazardous waste:
Users are required to read the MSD Chemical Hygiene Plan and relevant MSDS. Argonne employees are required to take the ESH-574 Chemical Waste Generator course as a procedural control. Non-Argonne users must develop a written waste-handling plan in consultation with the Lab Manager (Jon Hiller) and the EMC safety representative (Russell Cook).

Acids and bases:
Users are required to read the MSD Chemical Hygiene Plan and relevant MSDS.

Nanoparticles dispersible in fluids:
The slurries are not currently available for use and are stored in a locked cabinet.

Cryogenic fluids:
The EMC provides PPE. Users are not allowed to work with cryogenic fluids unless they also have trousers without cuffs and closed toe shoes with nonabsorbent uppers.

Elevated pressures and compressed gases:
Gas cylinders are only handled by EMC staff who have completed the ANL compressed gas cylinder course.

High temperatures:
Users are instructed not to bring any flammable chemicals near the hot plate even if it is off. Users should exercise caution when working near the hot plate when it is on. The potential for a burn will only occur if someone places exposed skin on the hot plate (i.e. you can unintentionally burn yourself).

Mechanical hazards:
Users are cautioned not to get their fingers between a vacuum sealing flange during pump down as a pinch of the skin could occur. Procedures include instructions to keep guards in place.

3.3 Personal Protective Equipment

List personal protective equipment (PPE) to be worn. Be specific to task or situation, unless it applies to all laboratory work in this project. Remember that safety glasses are not sufficient splash protection against certain chemicals. For gloves, be specific as to type appropriate for the task.

Task/Situation	Personal Protective Equipment
All laboratories	Safety glasses (ANSI Z87.1 compliant)
Cryogenic Handling	Section 4.10.10 of the ES&H Manual requires: • Cryogenic gloves (blue gloves provided by EMC) • Full face shield and safety(provided by EMC) • Trousers without cuffs • Shoes: closed-toe with non-absorbent material uppers • Lab coat with buttons fastened (or long-sleeve shirt with cuffs buttoned and the shirt tail outside the trousers).
Vacuum systems	Nitrile gloves (not to protect personnel but to prevent contamination of vacuum systems with body oils)
Chemical Handling	Nitrile gloves
Acids/Bases	Nitrile gloves, acid proof smock and full face shield.

3.4 Training

Indicate the training required for participation in this project. Include Argonne-supplied training (list course numbers and titles), job-specific training (indicate who provides training, how records are kept), and external training (academic requirement, specialized training and/or certification).

Project participants are trained by EMC staff for each activity and to operate each instrument by following specific Standard Operating Procedures (SOP). Safety has been incorporated into the SOPs by restricting the users to certain operations, by written/posted warnings, and by written instructions for off-normal or emergency situations. Trainees must go through a rigorous period of familiarization under the supervision of an authorized EMC staff member and demonstrate that they can operate each instrument according to its SOP before they are allowed to carry out a process or use a specific instrument without direct supervision.

Jon Hiller maintains lists of participants who are trained in the use of the instruments.

The required Argonne courses for this project are:

1. EMC101, EMC Users Orientation; available on-line.
2. ESH100U, ANL User Facility Orientation (or ESH100); available on-line.
3. ESH223U, Cyber Security Program Training (or ESH223); available on-line.
4. ESH377, Electrical Safety Awareness; available on-line.
5. ESH574, Chemical Waste Generator; required of those Argonne employees who generate hazardous wastes or waste oils.

Project participants are also required to read the MSD Chemical Hygiene Plan. Non-Argonne users must develop a written waste-handling plan in consultation with the Lab Manager (Jon Hiller) and the EMC safety representative (Russell Cook).

3.5 Chemical Storage

Describe the specific locations where chemicals and gases are stored including type of storage (e.g., flammables cabinet) and how hazardous chemicals are labeled. Include precautions taken for the storage of carcinogens. Indicate who is responsible for keeping the Chemical Management System (barcodes) up-to-date. Attach Material Safety Data Sheets (MSDS) for the particularly hazardous chemicals, and describe where all MSDS are available near the location of the project. The PI must ensure that MSDS for all chemicals used in this project are on file in the Chemical Management System (https://webapps.inside.anl.gov/cms/msds/) and that all participants have ready access to them.

Flammable chemicals are stored in a flammables rated cabinet in DL-126. Most of the chemicals are labeled from the manufacturer and stamped with the necessary precautions. Those chemicals that are transferred to another container are labeled with the chemical name (not chemical formula), and are labeled with the 4 diamond hazard label. Small transfer bottles of organic solvents are made available to the users and they are located on the laboratory workbench. These bottles when not used are stored in a tray to contain spillage. Under no circumstances are non-flammable chemicals and waste to be stored in the flammables cabinet.

Other types of chemicals and polishing media (diamond slurries, etc) are non hazardous and are located in the cabinets on the north and east walls. They are also labeled by the manufacturer.

Nanomaterial slurries are stored in a locked cabinet in DL126.

Argon cylinders are stored outside of DL126. The small cylinders of specialty gases used in the SPI Plasma Prep are stored under the bench on which that instrument sits.

Jon Hiller is responsible for maintaining the CMS and ordering of all chemicals that come into the lab. Users are restricted from bringing their own chemicals into the lab.

All MSDS are located in binders on top of the flammables cabinet and organized alphabetically by chemical name. All users of DL126 know where to find the MSDS. Jon Hiller also keeps an record of all MSDS on his office computer in A-257.

3.6 Sample Storage and Disposition

Describe how and where samples utilized and produced in this project are stored (and labeled!) while not in active use, how long they will be retained, and how they will be disposed of.

All users will remove their samples from the facility once they are done preparing them. The EMC does not store samples in the sample preparation lab. The exception to this is samples that are those of EMC staff members, where the samples are analytical standards or samples that are a work in progress for an active project. These samples are labeled with the date, sample composition, person it belongs to and possibly a unique code that the researcher assigns to it. The analytical standards will stay within the EMC. The other samples will be relocated once the project is completed.

The participants are responsible for removing their specimens and materials from the EMC. Most of those samples and materials are not hazardous.

3.7 Waste Handling

For all hazardous, radioactive, and mixed wastes, describe where and how they are accumulated (include satellite waste area number), and who will be responsible for writing up the waste for disposal by Waste Management. Indicate any special circumstances (special containers, venting, etc.) regarding the safe storage of waste. Address the prevention of incompatible waste mixtures. Include plans for dealing with the waste produced by the future decommissioning of equipment and termination of projects.

There will be no radioactive waste generated in the EMC. Jon Hiller is the custodian for the Satellite Waste Accumulation Area # 21200006 and is responsible for the proper disposal of all waste in DL-126. The lab generates about 1-2 gallons/year of waste pump oil (“special waste”), 1 gallon/year of acetone, 1 gallon/year of methanol, 1 gallon/year of other alcohols used for cleaning., and 2 cubic feet/year of towels soaked with oil or acetone or methanol.

Participants are instructed on how to log and place chemical waste in the Satellite Waste Accumulation Area (SWAA) during the training for each procedure.

The wastes created from acids and bases are stored where the acid or base stock is located and the waste is removed after sample preparation. Less than 1 liter of both acid and base waste is generated per year.

3.8 Emergency Management

If this project involves chemical hazards, esp. the use of corrosive chemicals, list locations of eyewash stations and safety showers. Indicate who is responsible for checking eyewash stations weekly.

There is one eyewash station located on the north wall of DL-126. Jon Hiller tests this eyewash on a weekly basis. All users are made aware of its location. The nearest safety shower is located on the east side of corridor D15 between DL136 and DL139. Building 212 maintenance is responsible for checking that shower.

Include emergency procedures in case of accidents, evacuations, or other hazardous situations. Include egress routes into common areas (hallways), safe shutdown procedures, and other pertinent information. Procedures may be attached. Are all hazard categories posted at the laboratory doors?

Standard building 212 evacuation procedures are in effect. All users are required to take EMC101 orientation which covers the evacuation process. Exit doors are posted. Hazard categories are posted on the laboratory door.

3.9 Additional Hazard Control

Describe here any measures of hazard controls that are not already documented in the previous sections.

N/A

3.10 Guidance Documents

List all documents, publications, and books, that you have consulted in the hazard analysis and control. Include relevant chapters and sections of the ES&H Manual but do not include those chapters that are requirements documents for other documents (e.g., 4.2). The divisional Chemical Hygiene Plan (http://www.msd.anl.gov/resources/esh/) is mandatory reading for all participants in projects that contain chemical hazards.

Hazard	Guidance Referenced
Toxic chemicals	ES&H Manual sections 4.1 & 4.3; MSD Chemical Hygiene Plan
Flammable chemicals	ES&H Manual sections 11.3 & 4.3
Carcinogenic chemicals	ES&H Manual section 4.5
Hazardous wastes	relevant MSDS; Argonne Waste Handling Procedures Manual; MSD Chemical Hygiene Plan
Strong acids or bases	ES&H Manual section 4.3; MSD Chemical Hygiene Plan; relevant MSDS
Cryogenic fluids	ES&H Manual section 4.10
High voltage equip.	ES&H Manual section 9.1
High vacuums	ES&H Manual section 13.1
Elevated pressures	ES&H Manual sections 13.1 & 4.3.11
Compressed gases	ES&H Manual sections 13.1 & 13.2
High temperatures	ES&H Manual section 11.2.15
Mechanical hazards	ES&H Manual section 7.15
Sharps	ESH Manual section 4.3.5
Nanomaterials	LMS Procedures 83 and 84

4. Working Within Controls (ISM Core Function 4)

4.1 List of Work Procedures

List all work procedures relevant to this project

Title	Date
Standard operating procedures (SOP) for each instrument or process	
Manufacturers' operating instructions	
Posted signs for PPE, lab hazards, etc.	
User's Guide to The EMC Specimen Preparation Laboratory	07/13/09
Posted lists of approved users	

4.2 Dosimetry

List locations where radiation dosimeters must be worn. Indicate if a ring is required in addition to the regular badge, and whether neutron dosimetry (type BGN) or not (type BG) is required. Consult with Health Physics regarding requirements.

Location	Dosimetry Requirement
N/A	

Identify individuals who will be issued dosimeters.

Name	Ring (Y/N)	Neutrons (Y/N)
N/A		

4.3 Safety Monitoring Equipment

Describe any equipment that is used to monitor safe working conditions (e.g., oxygen monitors, background radiation alarms). Note that all such equipment must be approved by Industrial Hygiene (or Health Physics for radiological monitoring).

N/A

4.4 Industrial Hygiene Monitoring

List the periodic Industrial Hygiene sampling that is required based on chemical, biological, or other hazardous materials used in this project.

N/A

4.5 Medical Surveillance

Identify individuals who will be placed in a medical surveillance program as a result of their participation in this project.

N/A

4.6 Working Alone

Indicate which tasks of this project are of sufficiently low hazard that they may be carried out by a participant working alone, in particular off-hours. Alternately, it may be more convenient to list the tasks that are prohibited while working alone. Note if different rules apply to specific qualification levels among the participants (e.g., students).

Prohibited working-alone tasks:

- Electropolishing, etching, or chemical polishing with acids/bases
- Mixing corrosives (acids and bases) to form a working solution

5. Feedback (ISM Core Function 5)

5.1 Records Kept

Identify types of records kept with this project that are useful in recreating and improving on the tasks within this project. In particular, include types of records that can be consulted if a task is unsuccessful or produces an unexpected result (in the scientific or operational sense). This could include lab notebooks, datasheets, computer data, instrument logs, images, etc.

Task/Situation	Record Kept
SWAA waste	Log is kept of chemical disposed and amount.
SWAA chemical inspection	Monthly inspection
Eye wash station	Weekly test log
Instrument performance	Instrument logs

5.2 Reporting

It is understood that technical results are reported to the outside world in scientific publications, presentations, and technical reports, and to the sponsor in program reviews, contractor meetings, and progress reports. Identify here the channels utilized to report the *operational* experience within the project, division, Argonne, or across the DoE complex. This should include emergency notifications, line

management notifications, Lessons Learned (good or bad), group meetings (may serve as pre- or post-job briefings) and other communication channels.

Emergency	1. Call 911. 2. Notify the laboratory supervisor, Jon Hiller (2-7904 or hiller@anl.gov). 3. Notify the EMC director, Dean Miller (2-4108 or miller@anl.gov). 3. Notify the 212 building manager, John Herman (2-6348 or 630-918-9784). 4. Notify the MSD division director, Michael Pellin (2-3510 or pellin@anl.gov). 5. Notify the MSD ESH coordinator, Urs Geiser (2-3509 or ugeiser@anl.gov).
Unplanned events or unexpected results that could affect worker safety and health, the environment, the general public, or Argonne's reputation	Stop work if danger is imminent. Immediately notify the laboratory supervisor, Jon Hiller (2-7904 or hiller@anl.gov).

6. Radioactive Materials Summary

This section to be filled out only if this project utilizes radioactive materials

What isotopes and amounts will be involved?

Isotope	Physical Form[a]	Total Quantity Involved[b]	Quantity Typically Used in a Single Experiment[b]

[a] Physical form could be salt, powder, liquid, gas, etc.

[b] Specify units (dis/min, Ci, etc.).

Provide a schedule for the necessary radiation monitoring.

Where will the experiment be performed? (Identify all laboratories to be used, as well as hoods and/or glove boxes.)

What special provisions will be made for waste disposal?

Are additional or modified emergency plans required? _____ Yes _____ No

If so, identify appropriate changes and additions.

Will the experiment involve special nuclear materials? _____ Yes _____ No

Has the appropriate signage for experimental areas been approved by Health Physics? _____ Yes _____ No

Have radiation monitors been ordered for all researchers? _____ Yes _____ No

Estimate the total external radiation dose equivalents from this work (in person-rems): _____

Health Physics Name Signature Date

7. Certification, Review and Approval

7.1 Certification

It is my belief that I have identified all the hazards relating to this work, and that by following the procedures outlined above the Materials Science Division and Argonne National Laboratory will be exposed to an acceptable level of risk. I will make this document available to all participants of the project.

J. M. Hiller 4/21/2008

Signature, Principal Investigator Date

7.2 Reviewers and Review Comments

List reviewers for this project and indicate (co-)coordinator/chair(s)

Igor Veryovkin 4/21/08	V. Novosad 4/21/08

Hazard level and review process used: High hazard/complexity _____ Low hazard/complexity ____

Review team comment

7.3 Environmental Compliance (NEPA)

The NEPA review is usually carried out in conjuction with the funding proposal that supports this project, prior to this safety review. Environmental Compliance Representative (ECR) comment:

No outstanding environmental issues

Urs Geiser Urs Geiser 4/21/2008

ECR Name Signature Date

7.4 Approvals and Authorization

The review team has reviewed the safety of this project and recommends its approval:

Chair 4/21/08 Co-chair 4/21/08

Chair/Co-chair signature Date Co-chair signature Date

Division director check one:

Approval of this project safety review authorizes this work to begin	✓
Separate work authorization is required (specify):	

I approve this project safety review:

George Crabtree George Crabtree 4-22-08

Division Director Signature Date

Included MSDS

Rather than attaching the MSDS for all of the potentially-hazardous chemicals and materials stored or used in DL126, the following pages list those materials. Items of particular concern are highlighted in red (acutely toxic OSHA-PEL and ACGIH-TLV values [< 1 ppm or < 0.5 mg/m^3]; "select" and Argonne Class 1 carcinogens; RCRA wastes). Acids and bases are flagged by green remarks.

ACGIH = American conference of Governmental Industrial Hygienists
OSHA = Occupational Safety and Health Administration
PEL = permissible exposure limit (most are TWA rather than STEL)
RCRA = Resource Conservation and Recovery Act
STEL = short term exposure limit
TLV = threshold limit value (most are TWA rather than STEL)
TWA = time weighted average

The chemicals in the following table are used regularly for the purpose of producing specimens for TEM or SEM from a wide variety of materials. Most laboratory users will encounter only a small number of these chemicals.

Material Name	OSHA-PEL	ACGIH-TLV	Carcinogen Classification	Remarks
Acetone	1000 ppm	500 ppm		RCRA waste: F003, U002, ignitable.
Aluminum oxide 0.5 & 3 µm powder	10 mg/m^3	10 mg/m^3		Component in Allied Hi Tech colloidal suspension.
Allied Hi Tech red lube diamond extender.	50 ppm	50 ppm		PEL/TLV based on ethylene glycol & ethylene glycol monobutyl ether components
Ammonium sulfate				Warm aqueous solution dissolves MgO.
Amyl acetate	100 ppm or 525 mg/m^3	50 ppm		Extender for silver paint. RCRA waste: ignitable.
Apiezon vacuum greases				
Argon gas				Simple asphyxiant used for ion milling.
Bon Ami glass cleaner				
Boron carbide powder				
Calcium sulfate dihydrate	15 mg/m^3 total dust, 5 mg/m^3 respirable fraction	10 mg/m^3 total dust containing no asbestos & <1% crystalline silica		Also sold under the name Drierite.
Carbon paint	see remarks	see remarks		The hazard varies with the manufacturer: it depends on

Material Name	OSHA-PEL	ACGIH-TLV	Carcinogen Classification	Remarks
				the solvent used. Bottles are marked. Solvents may be RCRA wastes.
Carbon tetraflouride + 10% Oxygen				Gas mixture for plasma etching glasses.
Cerium (IV) oxide 0.5 & 3 µm powder	10 mg/m^3	10 mg/m^3		Component in Allied Hi Tech colloidal suspension.
Citric acid anhydrous				**Acid.**
Crystalbond 509	1 ppm	1 ppm		PEL/TLV based on one component: pthalic anhydride.
Crystalbond 555	1 ppm		Select Carcinogen. Argonne Class 1	PEL & carcinogen ratings based on 1 component: ethylene oxide.
Crystalbond 562				
EFFA Duster				dichlorodiflouromethane, CAS 75-71-8
EPO-TEK 353ND Parts A & B				Specialized epoxy glue for specimen preparation.
H. B. Fuller epoxy resin				General-purpose glue.
Ethyl alcohol	1000 ppm	1000 ppm		RCRA waste: ignitable.
Ethylene glycol	50 ppm	50 ppm		Used for some grinding slurries.
Glycerin	15 mg/m^3 total dust, 5 mg/m^3 respirable fraction	10 mg/m^3		Used for some grinding slurries.
Hydrochloric acid	5 ppm	2 ppm		**Acid.**
Isopropyl alcohol	400 ppm	200 ppm		RCRA waste: ignitable.
Lapmaster abrasive polishing liquid				
LECO diamond suspension				
Loctite 420		0.2 ppm		Ethyl cyanoacrylate.
M-Bond 610 epoxy	200 ppm	200 ppm		PEL/TLV based on one component: tetrahydrofuran.
Methyl alcohol	200 ppm	200 ppm		RCRA waste: F003, U159, ignitable.
Methyl ethyl ketone				RCRA waste: F005, U159,

Material Name	OSHA-PEL	ACGIH-TLV	Carcinogen Classification	Remarks
				D035 (TCLP > 200 mg/l), ignitable.
MICRO cleaning solution				
Mineral oil	5 mg/m^3 (mist)	5 mg/m^3 (mist)		RCRA waste: "special".
Neolube #1 dry film lubricant	400 ppm	200 ppm		PEL/TLV based on one component: isopropanol. RCRA waste: ignitable.
Nitric acid	2 ppm	2 ppm		**Acid.**
Nitrogen gas				Used for backfilling vacuum chambers & ion-milling diamond.
Nitrogen liquid				
Old Dutch Cleanser				
Ortho-phosphoric acid	1 mg/m^3	1 mg/m^3		**Acid.**
Oxygen gas				
Potassium hydroxide		2 mg/m^3		**Base.** Used for etching Si.
1,2 Propanediol (propylene glycol)				Used in Allied Hi Tech diamond suspensions.
Silicon carbide powder	15 mg/m^3	10 mg/m^3		Used in slurries for cutting.
Silicon dioxide amorphous powder	20 M particles/ft^3	2 mg/m^3		Component in Allied Hi Tech colloidal suspension.
Sodium chloride				
Sodium hydroxide	2 mg/m^3	2 mg/m^3		**Base.**
SPI silver paint	0.01 mg/m^3	0.1 mg/m^3		PEL/TLV from one component: silver. RCRA waste: D011 (TCLP > 5 mg/l), ignitable.
Struers DP-Suspension				Diamond suspension for grinding & polishing.
Struers DP-Lubricant Green				Lubricant for grinding & polishing.
Struers cutting liquid for Accutom	5 mg/m^3 (mist)	5 mg/m^3 (mist)		Oil. RCRA waste: "special".
Sulfuric acid	1 mg/m^3	1 mg/m^3	Mist is a Select Carcinogen & Argonne Class 2.	**Acid.**

Material Name	OSHA-PEL	ACGIH-TLV	Carcinogen Classification	Remarks
Tetramethylammonium hydroxide				**Base.** Used to dissolve PMMA.
Three-in-One Oil	1000 ppm	1000 ppm		PEL/TLV from one component: ethanol. RCRA waste: "special".
Toluene		100 ppm or 375 mg/m^3		RCRA waste: F005, U220, ignitable.

The chemicals in the following table are used rarely and only in the production of TEM specimens by microtoming. These chemicals are not normally stored in DL126; rather, they are ordered and then used-up.

Material Name	OSHA-PEL	ACGIH-TLV	Carcinogen Classification	Remarks
Araldite GY-502 epoxy resin	5 mg/m^3	5 mg/m^3		PEL/TLV based on one component: 1,2 Benzenedicarboxylic acid dibutyl ester.
DER-736 epoxy resin				Polymer of epichlorohydrin-polyglycol, CAS 041638-13-5
DMAE epoxy resin				2-dimethylaminoethanol, CAS 108-01-0
DMP-30 epoxy resin				primary component is Tris-2,4,6-(dimethylaminomethyl) phenol, CAS 90-72-2
Dodecenyl succinic anhydride				
ERL-4206 epoxy resin	10 ppm	0.1 ppm	Argonne Class 2	Vinyl cyclohexene dioxide CAS 106-87-6
Nonenyl succinic anhydride (NSA)				CAS 28928-97-4

The chemicals in the following table are used rarely and only by select EMC staff for the purpose of producing analytical standards, or they are materials that are essential for EMC staff to maintain the operation of the center. Other laboratory users will not be using them.

Material Name	OSHA-PEL	ACGIH-TLV	Carcinogen Classification	Remarks
Beryllium	0.002 mg/m^3 (Be dust)	0.002 mg/m^3 (Be dust)	Be dust is a Select Carcinogen. Argonne Class 1	RCRA waste: P015 (acutely hazardous); bulk Be is in DL126 in a sealed container.
Chromium	1 mg/m^3	0.5 mg/m^3		RCRA waste: D007 (TCLP >

Material Name	OSHA-PEL	ACGIH-TLV	Carcinogen Classification	Remarks
				5 mg/l).
Copper	0.1 mg/m^3 (Cu fumes)	0.1 mg/m^3 (Cu fumes)		Bulk Cu is in DL126 and fumes are never created.
Dialac-C oil	5 mg/m^3	5 mg/m^3		Oil used solely for FEI & Philips microscopes. RCRA waste: "special".
Exxon Univolt 60 Oil	5 mg/m^3 (mist)	5 mg/m^3 (mist)		Electrical insulating oil used in AAEM. RCRA waste: "special".
Gadolinium				
Hafnium	0.5 mg/m^3	0.5 mg/m^3		
Magnesium				
Mica	20 M particles/ft^3			
Mikro-Kleer metal spray				
Molybdenum oxide	5 mg/m^3	5 mg/m^3		
Nickel oxide	1 mg/m^3	1 mg/m^3	Select Carcinogen. Argonne Class 1	NiO
Pfeiffer P3 vacuum oil	5 mg/m^3 (mist)	5 mg/m^3 (mist)		Oil for turbo pump bearings. RCRA waste: "special".
Phenanthrene	0.2 mg/m^3			Used in a sealed cartridge in the Zeiss FIB/SEM (DL135).
Santovac 5	5 mg/m^3 (mist)	5 mg/m^3 (mist)		Diffusion pump oil. RCRA waste: "special".
Selenium powder/pellets	0.2 mg/m^3 (Se powder)	0.2 mg/m^3 (Se powder)		RCRA waste: D010 (TCLP > 1 mg/l).
Silicon	15 mg/m^3 total dust, 5 mg/m^3 respirable fraction	10 mg/m^3		
Silicon monoxide				PEL/TLV based on one component: silica.
Smooth-On PC Series Epoxy	10 mg/m^3	6 mg/m^3		
Solvent-refined neutral paraffinic oil	5 mg/m^3 (mist)	5 mg/m^3 (mist)		Brands: Edwards High Vacuum, Inland Vacuum, etc; for roughing pumps. RCRA waste: "special".
Tungsten		5 mg/m^3		

Material Name	OSHA-PEL	ACGIH-TLV	Carcinogen Classification	Remarks
Tungsten hexacarbonyl				Used in a sealed cartridge in the Zeiss FIB/SEM (DL135).
Victawet				
Wenol metal polish		[a] 100 ppm [b] 5 mg/m^3		Also POL metal polish. [a] Aluminum oxide [b] Iron oxide (trace)
Yttrium barium copper oxide		0.5 mg/m^3		
Zinc				
Zirconium				